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METHOD OF MEASUREMENTS FOR PROPERTIES OF GYROMAGNETIC MATERIALS FOR USE AT MICROWAVE FREQUENCIES

PART III PERMITTIVITY, APPARENT DENSITY AND **CURIE TEMPERATURE**

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METHOD OF MEASUREMENTS FOR PROPERTIES OF GYROMAGNETIC MATERIALS FOR USE AT MICROWAVE FREQUENCIES

PART III PERMITTIVITY, APPARENT DENSITY AND **CURIE TEMPERATURE**

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METHOD OF MEASUREMENTS FOR PROPERTIES OF GYROMAGNETIC MATERIALS FOR USE AT MICROWAVE FREQUENCIES

PART III PERMITTIVITY, APPARENT DENSITY AND CURIE TEMPERATURE

0. FOREWORD

- **0.1** This Indian Standard (Part III) was adopted by the Indian Standards Institution on 22 April 1977, after the draft finalized by the Magnetic Components and Ferrite Materials Sectional Committee had been approved by the Electronics and Telecommunication Division Council.
- **0.2** With the increasing use of ferrites in electronics and telecommunication equipment, and their availability from indigenous manufacturers, it has become necessary to formulate a series of Indian Standards to establish measuring methods for their properties.
- **0.3** The object of this series of standards is to establish measuring methods for properties of gyromagnetic materials for use at microwave frequencies. The methods described herein do not exclude the use of other methods giving substantially the same or better results and accuracy.
- **0.4** This standard (Part III) is one of the series of Indian Standards relating to methods of measurements for properties of gyromagnetic materials for use at microwave frequencies. A list of standards of this series is given in Appendix A.
- **0.5** In preparing this standard, assistance has been derived from IEC document 51(C.O.)164 and 51(Sectt)140 'Draft measuring methods for properties of gyromagnetic materials intended for application at microwave frequencies' issued by the International Electrotechnical Commission.
- **0.6** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS: 2-1960*.

1. SCOPE

1.1 This standard (Part III) describes method of measurements of complex permittivity ϵ_r ; apparent density ρ_{app} ; and Curie temperature θ_c ; of ferrite materials for application at microwave frequencies.

^{*}Rules for rounding off numerical values (revised).

Note 1 — For the purpose of this standard, the words 'ferrite' and 'microwave' are used in a broad sense:

- by 'ferrites' are meant not only magnetodielectric chemical components having a spinel crystal structure, but also materials with garnet and hexagonal structures; and
- the 'microwave' region is taken to include wavelength between 1 m to 1 mm, roughly, the main interest being concentrated on the region 0.3 m to 10 mm.

Note 2 — Examples of components employing microwave ferrites are non-reciprocal devices, such as circulators, isolators and non-reciprocal phase-shifters. These constitute the major field of application, but the materials may be used in reciprocal devices as well, for example, modulators and (reciprocal) phase-shifters. Other applications include gyromagnetic filters, limiters and more sophisticated devices, such as parametric amplifiers.

2. DEFINITIONS

2.1 For the definitions of general terms used in this document, reference should be made to IS: 1885 (Part XXXI) - 1971*.

SECTION I METHOD OF MEASUREMENTS FOR COMPLEX PERMITTIVITY ϵ_r

3. SCOPE

3.1 This section describes the method of measurements for complex permittivity $\epsilon_{\mathbf{r}}$ of ferrite materials, for application at microwave frequencies.

4. METHOD OF MEASUREMENTS

- 4.1 Introduction A knowledge of the complex permittivity of a ferrite material is of primary importance for the theoretical analysis of wave propagation in ferrites as well as in the design of ferrite microwave components. Microwave ferrites frequently exhibit very low dielectric loss, in fact so low that it becomes difficult to measure, primarily due to the fact that it becomes difficult to discern between dielectric and magnetic loss. This difficulty is often avoided by subjecting the test sample to a very strong magnetic field; so strong that it saturates the material and moves the gyromagnetic resonance frequency well above the measuring frequency. By this means neither low-field nor resonance magnetic losses will be present to obscure the result.
- **4.2 Object** A method for the measurement of complex permittivity of isotropic ferrites for microwave applications by means of a resonant cavity is described. In this measurement a rod-shaped test specimen is coaxially arranged in a cylindrical cavity with openings in the wall to accept the ends of the specimen. The theory on which this method is based, is exactly valid only for the impracticable arrangement shown in Fig. 1, with no openings in the wall of the cavity and no air gap between the ends of the

^{*}Electrotechnical vocabulary: Part XXXI Magnetism.

specimen and the cavity; hence there will be a systematic error which may be only roughly estimated. However, as it is possible to keep the error approximately constant, it does not normally affect comparative measurements on different materials.

The cavity is designed for a measuring frequency of about 10 GHz, the actual value depending upon the characteristics of the specimen. This is, however, relatively unimportant, since the dielectric properties of ferrites change very slowly with frequency in the microwave region.

4.3 Theory — If an isotropic dielectric medium having an applied steady electric field strength E the electric displacement D is given by the equation:

$$D = \overline{\epsilon} \epsilon_0 E \qquad \dots \dots (1)$$

where ϵ_0 is the electric constant and ϵ is the relative permittivity.

If the medium is subjected to an alternating electric field, the electric displacement is not necessarily in phase with the field strength. This fact may be expressed mathematically by making ϵ a complex quantity. If we write $\epsilon = \epsilon' - j \epsilon''$, the imaginary part ϵ'' may be taken to represent the dissipation in the medium.

A cylindrical E_{010}^- resonator and a rod-shaped specimen are used for the measurement. Quantities that should be measured are the resonance frequency and loaded Q of the cavity with and without the specimen and the cavity and specimen dimensions. The method is not suitable for materials with dissipation factors tan $\delta = \frac{\epsilon''}{\epsilon'} > 0.1$.

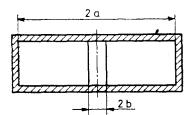


Fig. 1 Ideal Resonant Cavity with Specimen, Used for Theoretical Calculation (Sectional View)

The cylindrical resonant cavity contains the coaxially mounted cylindrical specimen. The permeability of the specimen μ' is approximately equal to 1 and the real part of the permittivity ϵ' is determined by the equation:

$$\sqrt{\epsilon'} \frac{\mathcal{J}_{1}\left(\frac{\omega}{c}\sqrt{\epsilon'}b\right)}{\mathcal{J}_{0}\left(\frac{\omega}{c}\sqrt{\epsilon'}b\right)} = \frac{\mathcal{J}_{1}\left(\frac{\omega}{c}b\right)\mathcal{N}_{0}\left(\frac{\omega}{c}a\right) - \mathcal{J}_{0}\left(\frac{\omega}{c}a\right)\mathcal{N}_{1}\left(\frac{\omega}{c}b\right)}{\mathcal{J}_{0}\left(\frac{\omega}{c}b\right)\mathcal{N}_{0}\left(\frac{\omega}{c}a\right) - \mathcal{J}_{0}\left(\frac{\omega}{c}a\right)\mathcal{N}_{0}\left(\frac{\omega}{c}b\right)} \dots (2)$$

 \mathcal{J}_0 and \mathcal{J}_1 being the Bessel functions of order, zero and one, \mathcal{N}_0 and \mathcal{N}_1 the Neumann-functions of order zero and one, c the velocity of light in free space, a the radius of the cavity, b the radius of the specimen (Fig. 2) and $\omega = 2\pi f$ the resonance angular frequency of the cavity.

Introducing f_0 , the resonance frequency of the empty cavity and f_1 , that of the cavity containing the specimen, the series expansion of equation (2) in ascending powers of $\delta f/f_0 = (f_0 - f_1)/f_0$ and b/a results in the following approximation, valid for small values of these quantities.

$$\epsilon' = \overline{\epsilon}' \left(1 - 0.722 \frac{b^2}{a^2} \overline{\epsilon}' \right) \qquad \dots (3)$$

with

$$\overline{\epsilon}' = 1 + \frac{0.78 \frac{\delta f}{f_0} \left(1 + \frac{0.692 \ a^2/b^2}{1 - 2 \ \delta f/f_0} \right)}{1 + 1.56 \frac{\delta f}{f_0} l_n \frac{a/b}{2.14}} \qquad \dots (4)$$

Assuming a lossless specimen ($\epsilon''=0$), yet with the real component of ϵ remaining unaltered, the change in 1/Q when substituting the lossless specimen by the real one is determined by the quotient of the following volume integrals:

$$\frac{1}{Q_1} - \frac{1}{Q_2} = \frac{\int_{\nu S}^{\kappa''} |E|^2 dV}{\int_{\nu C}} \qquad (5)$$

with

 $Q_1 = \text{loaded } Q \text{ with real sample,}$ $Q_2 = \text{loaded } Q \text{ of the cavity with lossless sample,}$

vs = volume of the sample,

vc = volume of the cavity with sample, and

E =amplitude of the electric field with lossless sample.

Using the same approximation as above this results in:

$$\epsilon' = \left(\frac{1}{Q_1} - \frac{1}{Q_2}\right) \left[\frac{0.27 \ a^2/b^2}{\left(1 - \frac{\delta f}{f_0}\right)^2 \left(1 + 1.56 \frac{\delta f}{f_0} \ln \frac{a/b}{2.14}\right)^2 + \epsilon' - 1}{1 - 1.45 \ \epsilon' \ b^2/a^2} \right] \frac{1}{1 - 1.45 \ \epsilon' \ b^2/a^2} \dots (6)$$

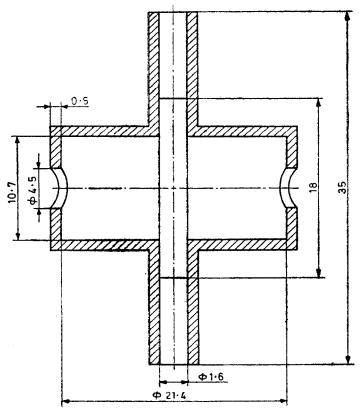
With the dimensions of Fig. 2, equations (4) and (6) become:

$$\epsilon' = \overline{\epsilon}' (1 - 4.0 \cdot 10^{-3} \overline{\epsilon}') = 1 + \frac{\frac{\delta f}{f_0} \left(0.78 + \frac{96.7}{1 - 2 \delta f / f_0} \right)}{1 + 2.86 \delta f / f_0} \dots (4a)$$

and

$$\epsilon'' = \left(\frac{1}{Q_1} - \frac{1}{Q_2}\right) \left[\frac{48\cdot 1}{\left(1 - \frac{\delta f}{f_0}\right)^2 \left(1 + 2\cdot 86\frac{\delta f}{f_0}\right)^2} + \epsilon' - 1 \right] \frac{1}{1 + 0\cdot 0081\,\epsilon'} \dots (6a)$$

with $Q_2 = Q_0 \left(1 + \frac{\delta f}{f_0} \cdot 0.65\right)$, Q_0 being the loaded Q of the empty cavity.



All dimensions in millimetres.

Fig. 2 Dimensions of the Resonant Cavity with Specimen;
Resonance Frequency of the Empty
Cavity 10.7 GHz

- 4.4 Test Specimen and Cavity The specimen shall be cylindrical, with a diameter of 1.60 ± 0.01 mm and a length of 18 ± 0.5 mm. It is inserted in a cylindrical transmission-type E_{010} cavity having dimensions according to Fig. 2. The ends of the specimen shall pass through holes in the cavity wall; the hole diameter being 1.64 ± 0.01 mm. The input and output lines of this cavity shall be made to appear as matched loads by the appropriate use of pads or isolators. The loaded Q of the cavity shall exceed 2 000. The test specimen shall be in a satisfactorily clean and dry state. There shall be a sufficiently strong axial magnetic field, as otherwise erroneous values of ϵ'' will be obtained due to the presence of magnetic loss.
- **4.5 Measuring Apparatus** Figure 3 is a schematic diagram of the equipment required for the measurement. Power from a suitable unmodulated or amplitude modulated microwave source A is run through a variable attenuator D and kept at a constant level throughout the measurement with the aid of a directional coupler E a crystal detector and a power-indicating meter F. This constant power is run through a precision variable attenuator G to the cavity H and the cavity output power is detected and indicated on a suitable meter I.
- **4.6 Measurement Procedure** Introduce an attenuation of 3dB with the precision attenuator. Without the specimen in the cavity, adjust the microwave frequency to cavity resonance, as indicated by maximum power output with respect to frequency variation. Note the output power level and measure the resonant frequency f_o with a wavemeter or other suitable means at B. Remove the 3dB of attenuation and locate the two frequencies at which the output power is the same as at cavity resonance with the 3dB attenuation in. Determine the separation in frequency of these two halfpower points at B by a heterodyning technique utilizing a frequency stabilized source C or any other technique giving sufficient accuracy. The loaded Q of the cavity is then given by:

$$Q_0 = \frac{f_0}{\Delta f_{1/2}}$$

where $\triangle f_{1/2}$ is the frequency separation of the half-power points.

Place the specimen in the cavity and measure f_1 and Q_1 in the same way. During the measurement, the specimen shall be in an axial magnetic field. The field strength shall be high enough for the measuring results to be independent of any further increase (normally above 400 kA/m). The measurements should be carried out at room temperature, approximately 25°C.

- **4.7 Calculation** Calculate the values of ϵ' and ϵ'' by means of equations (4a) and (6a) and the dielectric loss factor $\tan \delta_e \epsilon'' / \epsilon'$.
- **4.8 Accuracy** The error of the approximation for ϵ' by equation (4a) is practically negligible.

The approximation for ϵ'' by equation (6a) gives a figure which is too high. The error is less than 3 percent, if $\delta f/f_0 < 0.17$, for example $\epsilon' < 16$.

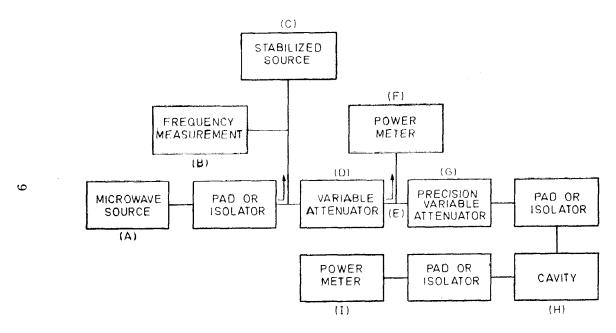


Fig. 3 Schematic Diagram of Equipment Required for the Measurement of Complex Dielectric Constant

The effect of the coupling holes is to increase the measured value of ϵ'' . This error is less than 1.5×10^{-4} for $\epsilon' < 16$. (For the influence of the magnetic loss see 4.4).

An error in the specimen diameter 2b (Fig. 1) results in a relative error in $(\epsilon'-1)$ and ϵ'' which is twice as high and has reciprocal sign, for example ± 1.3 percent with $b=1.60\pm0.01$ mm.

Due to the influence of the holes in the walls of the cavity (into which the ends of the specimen protrude), the measured value of ϵ' becomes too small.

4.9 Data Presentation — The report shall include the following:

- a) Values of ϵ' , ϵ'' and $\tan \delta_e$; and
- b) Temperature of the material during the measurement and unique identity of specimen.

SECTION 2 METHOD OF MEASUREMENTS FOR APPARENT DENSITY ρ_{ann}

5. SCOPE

5.1 This section describes the method of measurements for apparent density ρ_{app} of ferrite materials, for application at microwave frequencies.

6. METHOD OF MEASUREMENTS

6.1 Introduction — Apparent density may be measured either by water densitometry or by mensuration. For porous materials (where the porosity is greater than 3 percent) mensuration yields a sufficient accuracy (±1 percent) and may be more convenient than water densitometry. For materials of low porosity (less than 3 percent) a more accurate estimate of apparent density is required and water densitometry is recommended.

6.2 Apparent Density (by Mensuration)

- **6.2.1** Object To measure the apparent density of porous polycrystalline gyromagnetic materials by mensuration. The method is applicable to materials whose porosity is greater than 3 percent by volume. It is also admissible for materials whose porosity is less than 3 percent by volume, but the alternative method as given in **6.3** is to be preferred on accuracy and economic considerations. The method given here is capable of accuracy better than +1 percent.
- **6.2.2** Theory Apparent density (ρ_{app}) of a standard test specimen is defined as the ratio of its mass to its volume.
- 6.2.3 Test Specimen The test specimen should be a regular, machined body of volume not less than 5 cm³. It may be either a right circular cylinder or a right angled parallelepiped. If a parallelepiped is employed it should ideally be a cube since for a given volume of sample machined to a given absolute tolerance the errors due to machining accuracy will then be

a minimum. The specimen should be cleaned by boiling in fresh methylene chloride for 3 minutes and subsequently dried for 30 minutes at approximately 110°C. Other cleaning methods capable of achieving the same result are also acceptable.

- **6.2.4** Measuring Apparatus A balance capable of weighing to an accuracy of ± 0.02 g is required together with a calibrated micrometer reading to ± 0.01 mm.
 - **6.2.5** Calibration The apparatus is assumed to be calibrated as in **6.2.4**.
- **6.2.6** Measuring Procedure The cleaned, degreased and dried specimen is weighed and its dimensions measured. Two independent weighings should be made and at least two independent estimates of each dimension obtained.
- **6.2.7** Calculation The apparent density of the test specimen is calculated as:

$$\rho_{\mathrm{app}} = W/V$$

where

W = weight of specimen, and

V= volume as calculated from the linear dimensions.

- **6.2.8** Accuracy The method is inherently beset by inaccuracies arising from random macroscopic defects in the specimen shape, particularly chipping along edges and at corners. If the sample is a cube of volume much less than 5 cm³ this source of error becomes serious and may be greater than ± 1 percent. Errors due to edge chipping may be minimized by increasing the cube volume, or alternatively by choosing a cylindrical shape of similar volume. In the cylindrical case the error may further be minimized by choosing the length to be large compared with the diameter. The mensuration method will always underestimate the true apparent density since it assumes that all surface defects are an intrinsic property of the test specimen.
- **6.2.9** Data Presentation Apparent density should be quoted as: 'Apparent density (by mensuration) xyz g/cm³±1 percent'.

6.3 Apparent Density (by Water Densitometry)

- **6.3.1** Object To measure the apparent density of dense polycrystalline gyromagnetic materials by water densitometry. The method is applicable to non-hygroscopic, dense ceramic materials whose porosity is lower than 3 percent by volume, and is capable of accuracy better than ± 0.2 percent.
- **6.3.2** Theory Apparent density (ρ_{app}) of a standard test specimen is defined as the ratio of its mass to its volume under standard conditions. An accuracy of the order of ± 0.2 percent is required if measurements of apparent density are to have any useful meaning, since real variations of more than 0.5 percent of the density frequently imply an intolerable variation in other properties of the material. Since there is no serious difficulty in

achieving weighings accurate to the order of ± 0.02 percent it is the measurement of volume which becomes the accuracy determining step. Provided, that the porosity is sufficiently low for the pores not to be interconnected, the most accurate and economic method of measuring volume, and hence the density, is by displacement of water. In this method the volume V of the sample is given by the difference in weights of the sample suspended in air and water;

thus $V = (W_1 - W_2)/(\rho_w - \rho_a)$

where

 $W_1 = \text{mass of sample suspended in air,}$

 W_2 = mass of sample suspended in water,

 $\rho_{\rm w}$ = density of water under the measurement conditions, and

 ρ_a = density of air under the measurement conditions.

The apparent density is then:

$$\rho_{\rm app} = \frac{W_1 \; \rho_{\rm w} - \; W_2 \; \rho_{\rm a}}{W_1 - \; W_2}$$

- **6.3.3** Test Specimen The test specimen should be a regular, machined body of volume not less than one cubic centimetre. The specimen should be cleaned by boiling in fresh methylene chloride for 3 minutes, and subsequently dried for 30 minutes at approximately 110°C. Other cleaning methods capable of achieving the same result are also acceptable.
- **6.3.4** Measuring Apparatus The measurement requires a balance capable of weighing to ± 0.001 g and a vessel containing distilled water. The aperture of the vessel should not be less than three times the maximum dimension of the test specimen.
- **6.3.5** Calibration The balance is assumed to be calibrated as given in **6.3.4**.
- **6.3.6** Measuring Procedure The cleaned and degreased test specimen is weighed in air (W_1) by suspending it from the balance beam by a thread whose mass is less than 0.02 percent of the expected mass of the test specimen. A human hair is most suitable. The specimen is then weighed (W_2) completely immersed in distilled water, care having been taken to ensure that all surfaces of the specimen are thoroughly wetted and that no air bubbles adhere. A small quantity of wetting agent may be added to the water if desired, and the temperature of the water should be noted.
- **6.3.7** Calculation The apparent density of the test specimen is calculated as:

$$\rho_{\rm app} = (W_1 \ \rho_{\rm w} - W_2 \ \rho_{\rm a})/(W_1 - W_2)$$

the values of $\rho_{\rm w}$ and $\rho_{\rm a}$ being obtained from standard tables.

6.3.8 Accuracy

6.3.8.1 Sources of random error include the following:

a) The failure of the water to wet the specimen surface adequately and

- the consequent adherence of air bubbles thereto; this leads to a random tendency to underestimate the density; and
- b) Inaccuracies in the weighings themselves. Reasonable precautions as outlined above will result in the sum of random errors being no greater than ± 0.1 percent.
- **6.3.8.2** The most significant sources of systematic error are:
- a) the presence of surface porosity in the test specimen, and
- b) incorrect values for ρ_w and ρ_a , the densities of air and water respectively.

Failure to allow for ρ_a results in a systematic error of order 0·1 percent and failure to allow for deviations of ρ_w from unity as the water temperature differs from 40°C results in a systematic error of about 0·02 percent per °C. Provided that the porosity is less than 3 percent the error due to exposed surface pores may be neglected beyond about 3 percent porosity the errors due to interconnection become significant and the method is no longer appropriate. For dense materials as defined in **6.3.1** above this method is advantageous over mensuration since the errors due to macroscopic surface irregularities are entirely avoided. The method as described is capable of accuracy typically ± 0.2 percent, as compared with ± 1 percent by mensuration of similar size specimen.

6.3.9 Data Presentation — The apparent density should be quoted as follows: 'Apparent density (by water densitometry) — xyz g/cm³ ± 0.2 percent'.

SECTION 3 METHOD OF MEASUREMENTS FOR CURIE TEMPERATURE, θ_c — Under Consideration

APPENDIX A

(Clause 0.4)

IS: 8426 Method of measurements for properties of gyromagnetic materials for use at microwave frequencies:

Part I Magnetization,

Section 1 Saturation magnetization, M_8

Section 2 Magnetization (at specified field strength), $M_{\rm H}$

Part II Resonance linewidth,

Section 1 Gyromagnetic resonance linewidth, \triangle H and effective lande factor, $g_{\rm eff}$ (general)

Section 2 Spin-wave resonance linewidth, $\triangle H_k$ Section 3 Effective resonance linewidth, $\triangle H_{\text{eff}}$

Part III Permittivity, apparent density and curie temperature,

Section 1 Complex permittivity ϵ_r

Section 2 Apparent density ρ_{app}

Section 3 Curie temperature $\theta_{\mathbf{c}}$

INDIAN STANDARDS

ON

MAGNETIC COMPONENTS AND FERRITE MATERIALS

1176-1969 Dimensions for aerial rods and slabs made of ferromagnetic materials 1885 (Part XII)-1966 Electrotechnical vocabulary: Part XII Ferromagnetic oxide materials 1885 (Part XXXI)-1971 Electrotechnical vocabulary: Part XXXI Magnetism 1885 (Part XLI)-1975 Electrotechnical vocabulary: Part XLI Non-reciprocal electromagnetic components 2032 (Part XVII)-1975 Graphical symbols used in electrotechnology: Part XVII Symbols for ferrite cores and magnetic storage matrices 6077 (Part I)-1971 Permanent magnets: Part I General requirements and tests 6235-1971 Dimensions of pot-cores made of ferromagnetic oxides and associated parts 7416 (Part I)-1974 Dimensions of TV ferrite components: Part I Cores for deflection coil 7416 (Part II)-1976 Dimensions of TV ferrite components: Part II Ferrite rod for linearity control unit 7416 (Part III)-1976 Dimensions of TV ferrite components: Part III Tuning magnet for linearity control unit 7416 (Part IV)-1976 Dimensions of TV ferrite components: Part IV Ring magnet for linearity control unit 7416 (Part V)-1976 Dimensions of TV ferrite components: Part V Segment magnet for linearity control unit 7416 (Part VI)-1976 Dimensions of TV ferrite components: Part VI Beam centering magnet for deflection coil 7416 (Part VII)-1976Dimensions of TV ferrite components: Part VII Pin cushion correction magnet for deflection coil 7416 (Part VIII)-1976 Dimensions of TV ferrite components: Part VIII U and I Core assembly for line output transformer 7416 (Part X)-1976 Dimensions of TV ferrite components: Part X Corner correction magnet 7416 (Part XI)-1976 Dimensions of TV ferrite components: Part XI Balun corner 7430-1974 Dimensions of screw cores made of ferromagnetic oxides 7431 (Part I)-1974 Tests for magnetic properties of ferrite aerial rods: Part I For long and medium wave receptions 7431 (Part II)-1976 Tests for magnetic properties of ferrite aerial rods; Part II For short

wave reception
7489-1974 Dimensions of cross cores (X-cores) made of ferromagnetic oxides and associated

parts

7527-1974 Dimensions of loudspeaker magnets

7616-1974 Guide for calculation of the effective parameters of magnetic piece parts

7687-1974 Methods of measurement for cores for inductors and transformers for telecommunications

7717-1974 General requirements and tests for magnetic cores for application in coincident current matrix stores having a nominal selection ratio of 2: 1 and in linear select memory stores